

MATERIALS SIG QUANTIFICATION AND CHARACTERIZATION
OF SURFACE CONTAMINANTS

Russ Crutcher
Boeing Defense and Space Group
Kent, Washington

ABSTRACT

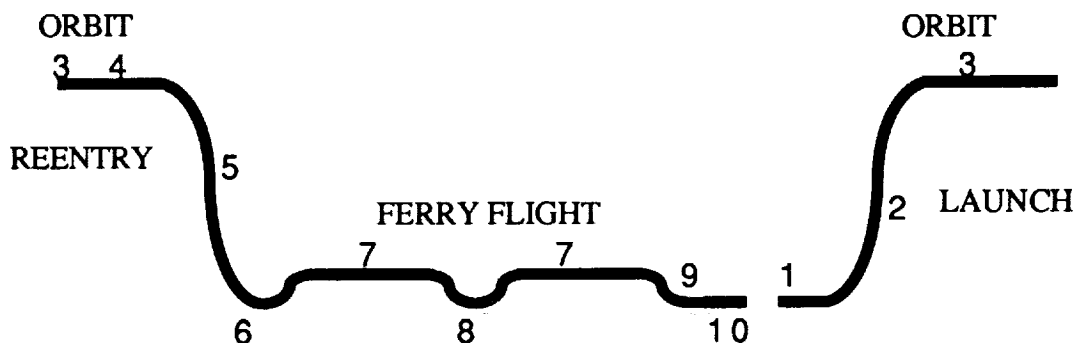
When LDEF entered orbit its cleanliness was approximately a MIL-STD-1246B Level 2000C. Its burden of contaminants included particles from every part of its history including a relatively small contribution from the shuttle bay itself. Although this satellite was far from what is normally considered clean in the aerospace industry, contaminating events in orbit and from processing after recovery were easily detected. The molecular contaminants carried into orbit were dwarfed by the heavy deposition of UV polymerized films from outgassing urethane paints and silicone based materials. Impacts by relatively small objects in orbit could create particulate contaminants that easily dominated the particle counts within a centimeter of the impact site.

During the recovery activities LDEF was 'sprayed' with a liquid high in organics and water soluble salts. With reentry turbulence, vibration, and gravitational loading particulate contaminants were redistributed about LDEF and the shuttle bay. Atomic oxygen weakened materials were particularly susceptible to these forces. The ferry flight exposed LDEF to the same forces and again redistributed contaminants throughout the bay.

Once in SAEF-2 there was a steady accumulation of particulate contaminants. These included skin flakes, paper fiber, wear metals, sawdust, and pollen to name a few. Some surfaces had a tenfold increase in their particle loading during their stay in SAEF-2. A few of the cleaner surfaces experienced a hundredfold increase.

INTERVALS IN THE HISTORY OF LDEF WITH DISTINCT CONTAMINATION ENVIRONMENTS

LDEF has been exposed to a variety of discrete environments over its lifetime. The prelaunch environment was a time when the new surface of trays, clamps, and the superstructure of LDEF were exposed to assembly debris, skin flakes, hair and fiber, insects, minerals, etc. The remnants or modified forms of these materials show the effects of exposure to all subsequent LDEF environments. The launch phase exposed LDEF to materials characteristic of the Shuttle Bay. These included bay liner materials, tile fiber and debris, and a variety of other contaminants common in the Shuttle bay but not unique to the bay. The launch environment is characterized by decompression, vibration, and a general acceleration force of about three times normal gravitation. These effects promote the migration of larger particles in the bay toward vents and toward surfaces that are normal to and face the acceleration vector. Once in orbit the environment is dominated by the orientation of the satellite with respect to the ram vector and atomic oxygen, to the thermal and ultraviolet light exposure, and to micrometeorite and debris impacts. Position four below marks the effects of grappling and docking operations. The first significant, although very low, acceleration loading after nearly six years in orbit occurred when the grapple arm attached to LDEF. Numerous objects, from as large as solar cell panel samples to small flakes of aluminum foil, began drifting from LDEF. Some relocation of materials from one LDEF surface to another probably occurred at this time. Reentry and landing exposed the orbitally degraded surfaces of LDEF to turbulent repressurization, acceleration and vibration loading, and to the reactive atmospheric gases, including water vapor. It also provided an environment in which cross contamination with the Shuttle Bay could occur. On the ground the Shuttle was exposed to natural minerals and other common airborne materials. The ferry flights exposed LDEF to decompression, repressurization, thermal cycling, and high humidity. Intervening stops during the ferry flight exposed LDEF to other contaminants. Once at Kennedy the ground operations prior to SAEF-2 exposed LDEF to a variety of particulate contaminants that were free of the effects of orbital exposure. Organic fibers, pollen grains, and insect debris were among the most obvious new contaminants. In SAEF-2 exposure to these types of materials continued with abraded floor materials, more pollens, skin flakes, and disassembly debris being added. The subject of this presentation is an overview of the changes in the contaminant distribution and character from grappling (4) to the final handling in SAEF-2 (10).



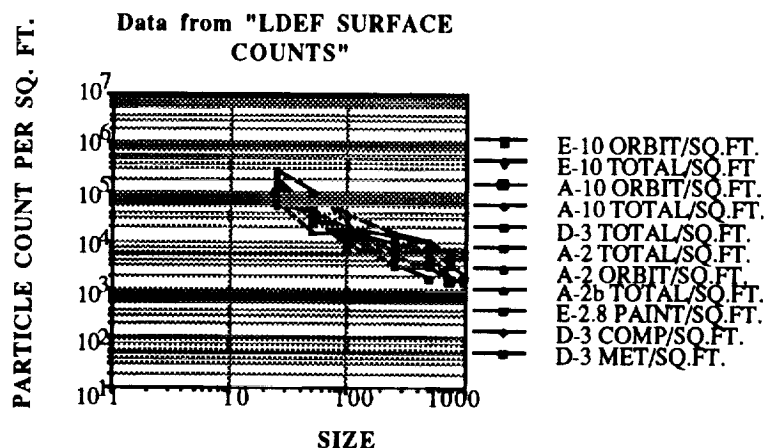
1. Condition of LDEF prior to launch: >MIL-STD-1246B, level 1000C for many trays.
2. During launch particulate contaminants are redistributed and Shuttle Bay debris is added.
3. Contaminants are modified and new contaminants are generated in the orbital environment.
4. Grappling jars particles and films free; some may have relocated on LDEF.
5. During reentry particles and brittle molecular contaminant films relocate.
6. The shuttle is exposed to the Edwards environment, accumulation of natural dusts.
7. High humidity, high gas flow velocities, thermal and pressure stresses occur.
8. HEPA filter fibers appear on tapelifts after exposure to new filters.
9. Ground operations prior to SAEF-2 include many manipulations to LDEF in complex environments.
10. SAEF-2 exposure.

Contamination Exposure History of LDEF

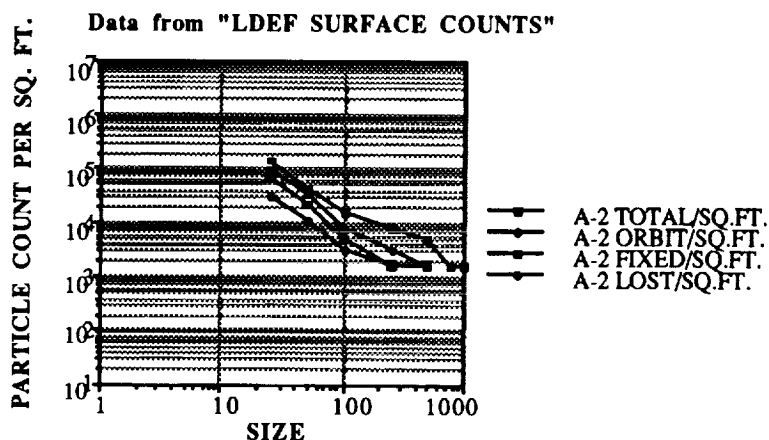
INCREASE IN SURFACE CONTAMINATION FROM ORBIT TO FINAL PACKAGING IN SAEF-2

On trays A-2, A-10, and E-10 particle counts were made on selected areas to determine the cleanliness of the surface in orbit, the migration of particles present in orbit during recovery operations, and the cleanliness as received from SAEF-2. The surfaces counted for each of these trays were silver backed Teflon. The particles present in orbit could be identified by the silhouette of the particle on the surface, indicating that the surface had been protected during its orbital exposure. For the leading edge trays exposed to a high fluence of atomic oxygen (A-10 and E-10) the silhouette was a small area of surface not eroded to the same extent as immediately adjoining areas. For tray A-2 the silhouette was the area protected from the brown returning molecular contaminants. All of the particles present during orbit were indicated by the term "Orbit". Some of the particles present in orbit were still present when the sample was analyzed. These were particles that had a silhouette of themselves on the surface beneath them. Such particles were indicated as "Fixed" particles. The count of particles actually present on the surface as received from SAEF-2 was indicated by the term "Total". The total count after SAEF-2 was from about two to four times the number of particles present on the surface during orbit.

The upper graph illustrates that the analyses of the particle population from Teflon surfaces, composite surfaces (D-3 COMP), metal surfaces (D-3 MET), and painted surfaces (E-2.8 PAINT) all seem to be within the same order of magnitude.



Particle Counts From LDEF Surfaces, Total Counts of all Particles and Counts Of Those Particles Present During Orbit.



Tray A-2 Detailed Particle Count of Total Particles, Those Present During Orbit, Those Fixed During Orbit and Still Present, and Those Present During Orbit Now Missing.

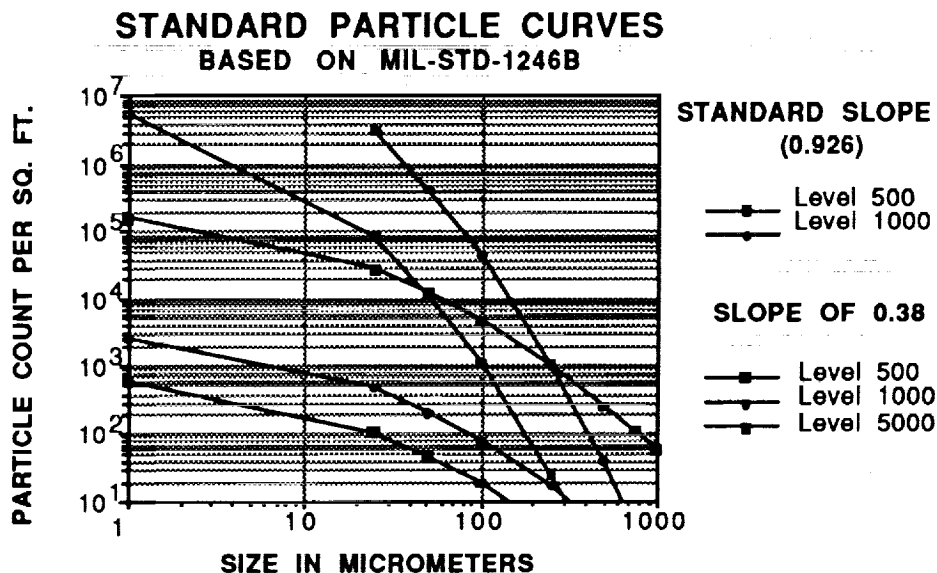
MIL-STD-1246B PARTICLE DISTRIBUTION CURVES AND LOG/LOG SQUARED CURVES WITH A DIFFERENT SLOPE

MIL-STD-1246B establishes cleanliness levels based on a particle size distribution assumed to be linear when the log of the cumulative number of particles greater than a specified diameter is plotted by the square of the log of the diameter. The standard further establishes the slope of the resultant line to be 0.926.

The log/log squared particle distribution with a slope of 0.926 was based on empirical data generated by measuring the removal efficiency by size for a standard material whose mass increased by the cube of the diameter. This is not the general case. The mass of a fiber varies linearly with its longest diameter. Pollens and spores decrease in density with increasing diameter. Skin cells have a mass that increases by the square of their diameter.

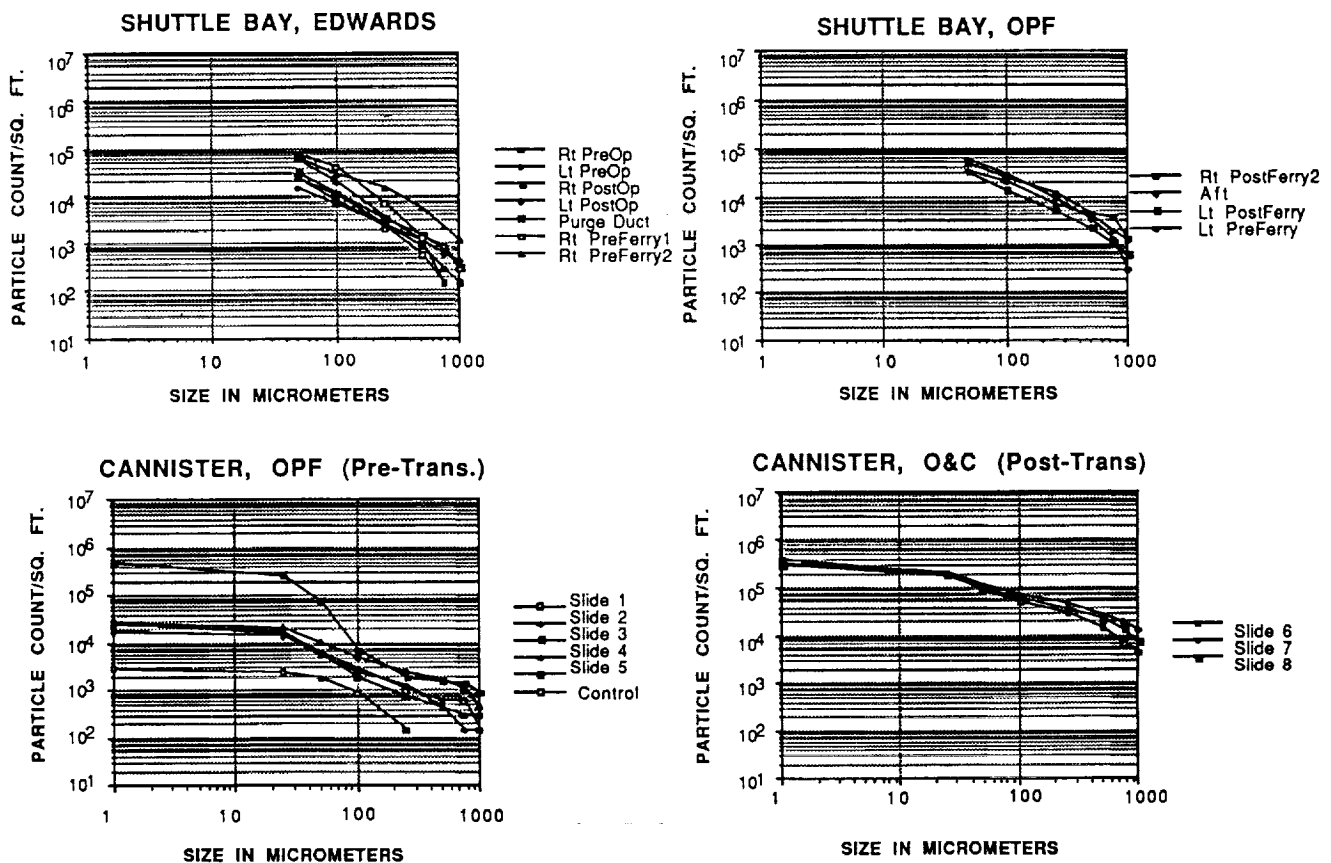
Pollens and fibers are not randomly sized but have specific dimensions characteristic of their origin. The 0.926 slope has a built in assumption that the particle population is the same for each size particle. The sedimentation rate for large particles is much greater than that for small particles; so even though there are more small particles, the large particle population becomes disproportionately represented on surfaces collecting particle fallout. As a result, though the log/log squared distribution still seems reasonable, the actual distribution seen on surfaces is often better characterized using an alternative slope. For many of the surfaces on LDEF slopes as low as 0.38 are indicated.

When identifying a cleanliness class using MIL-STD-1246B some arbitrary sized particle must be selected to establish the level if the particle distribution curve does not have a slope of 0.926. A particle distribution with a slope of 0.38 and one particle per square foot greater than 5000 micrometers could be assigned a cleanliness level of 1000 for particles less than 250 micrometers or a level 500 based on particles smaller than 50 micrometers.



PARTICLE DISTRIBUTION CURVES AND LDEF ASSOCIATED PARTICLE COUNTS

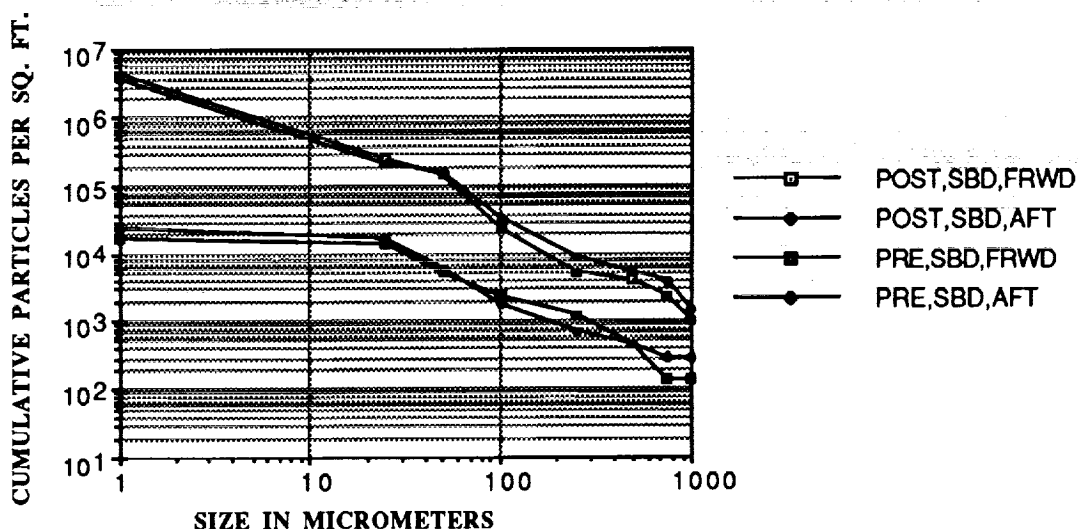
These charts are based on the results of tapelift samples from the Shuttle Bay and the Transportation Canister collected when LDEF was located within each respective container. The particle count distribution by size curves for the Shuttle Bay and for the Transportation Canister are much more shallow than the 0.926 slope used for the MIL-STD-1246 curves. The Shuttle Bay samples collected in the OPF are very close to a slope of 0.38. All of the particle count data for LDEF is shown in graphical form. The graphical format is used because the particle distribution is not conducive to the assigning of a MIL-STD-1246B cleanliness level. The significance of a list of numbers is also less informative than seeing the shape of the distribution. Unusual distributions such as that in the pre-transportation Transportation Canister sample are easily seen in a graphical format.



DISTRIBUTION OF PARTICLES IN THE TRANSPORTATION CANISTER BEFORE AND AFTER TRANSPORTING LDEF

The Transportation Canister was relatively clean prior to transporting LDEF from the OPF to the O&C building based on tapelift samples collected from the floor of the Canister. Most of the particles were small metal fragments. Many of these were in a line as a result of scratches on the surface of the Canister floor. Some of these fragments were bound together with an organic binder. Skin particles, paper fiber, clean room wiper residue, starch grains, sand grains, and vinyl flooring residue were also present. After transporting LDEF the particle count increased by nearly an order of magnitude or more. The second set of lifts were collected from approximately the same location as the first set. LDEF debris was a major reason for the increase in the number of particles but other sources also made a significant contribution. The LDEF debris was identifiable as very thin metal foils, Kapton particles, and fine ash particles. These materials accounted for over half of the increase. The balance was spray paint residues, paper fibers, calcite, starch, soil particles, pine pollen, and rust. The size distribution of the LDEF debris ranged from submicrometer to millimeters in greatest dimension. The non-LDEF debris was predominantly between five and one hundred micrometers with some of the fibers exceeding the millimeter range in length. The proportion of LDEF debris to other contaminants was smaller than had been expected. This may have been due to much of the more easily removed LDEF debris having been already removed by the earlier activities or having been moved to locations on LDEF where they were stable with LDEF in the fixed, row 12 top, configuration. The accumulation of more contaminants on the Canister floor not directly attributable to LDEF suggests that the upward facing surfaces of LDEF would receive contaminants from the Canister cover. These contaminants would include paper fibers, pollens, etc. The two plots of the particle size distribution before transporting LDEF are very close to one another, as are the two plots for the samples collected after transportation. This would seem to indicate that this was not a localized effect but was representative of what occurred during transport.

**TAPELIFT COUNTS FROM THE TRANSPORTATION
CANISTER BEFORE AND AFTER TRANSPORT OF LDEF**

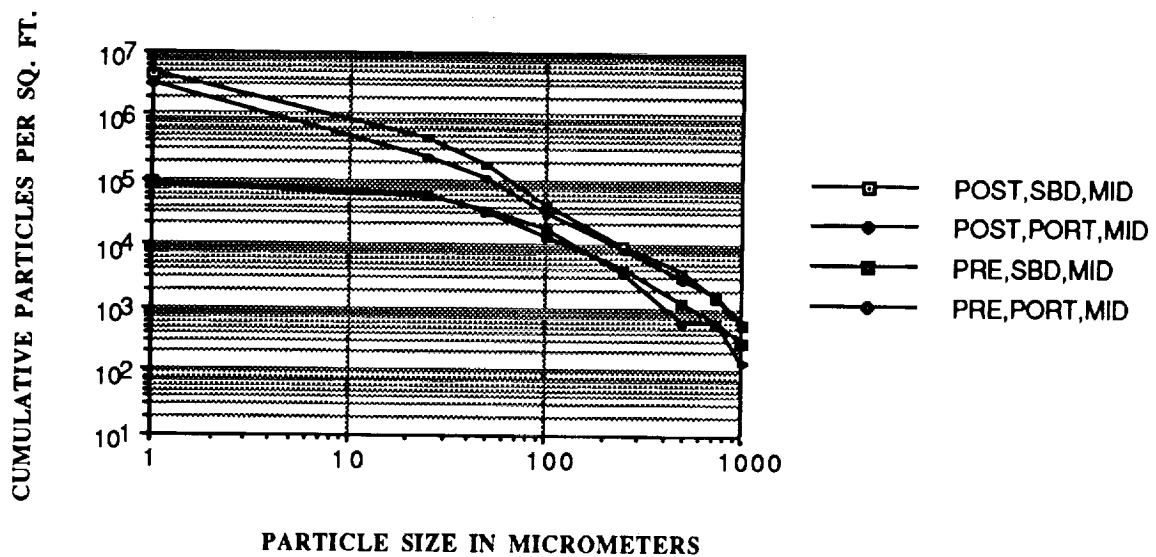


DISTRIBUTION OF PARTICLES IN THE LATS BEFORE AND AFTER TRANSPORTING LDEF

The LATS was not nearly as clean as the Transportation Canister before the transport of LDEF. Tapelift samples collected from the port and starboard sides of the LATS floor near the middle of LATS had particle counts that were a factor of ten greater than was found on the pre-transport Transportation Canister for particles smaller than one hundred micrometers.

After transport of LDEF the particle size distribution curves for the LATS samples were nearly the same as the post-transport samples from the floor of the Transportation Canister. The contribution from LDEF however was much less. Less than a third of the increase was due to LDEF particles. Cleaning residues, spray paint residues, pollens, insect parts, paper and clothing fiber, and black foam particles were more common. The LATS activities probably contributed more new contaminants to LDEF than did the Transportation Canister.

TAPELIFT COUNTS FROM THE LATS BEFORE AND AFTER TRANSPORTATION OF LDEF TO SAEF-2

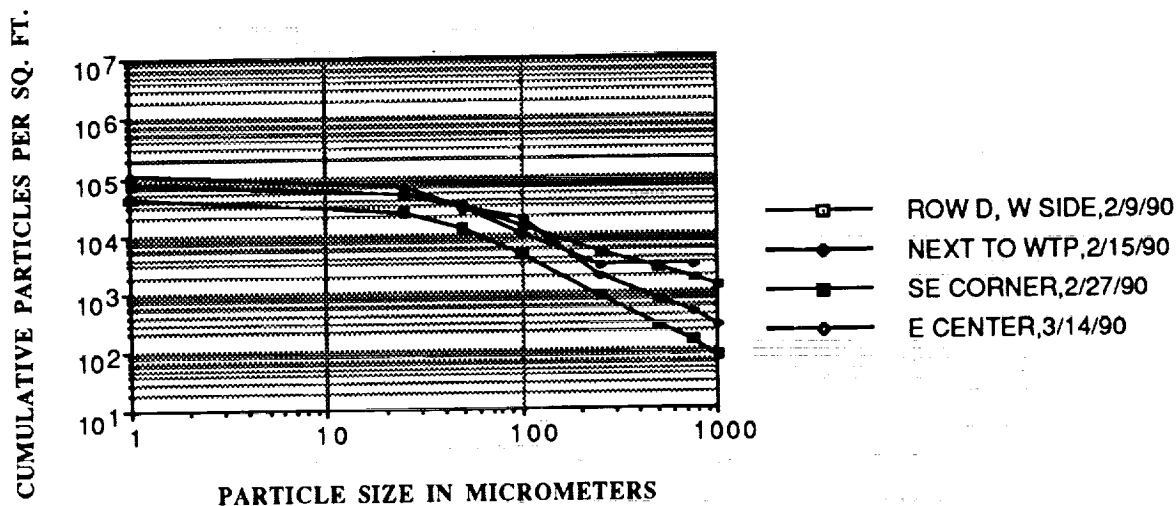


SURFACE CLEANLINESS OF LATS BASED ON PARTICLE FALLOUT PAD AND TAPELIFT ANALYSIS IN SAEF-2

The floor of the LATS was cleaned regularly (daily) to reduce the opportunity for the mechanical transport or lofting of debris to LDEF. The tapelift samples were collected at midday on the days noted. The surfaces sampled were areas of low traffic. The sample collected on the fifteenth of February was taken adjacent to one of the fallout pads. These samples indicate a relatively low cleaning efficiency and a rapid sedimentation rate.

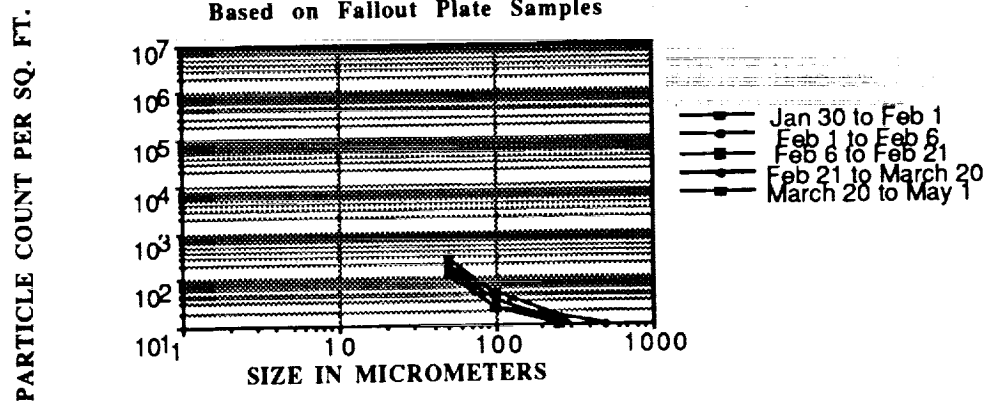
The fallout pad data should be lower than the tapelift results in that the fallout pad collects only fallout and not mechanically transferred debris, but the difference in these two plots indicates that the fallout pad data grossly underestimates LDEF's exposure. Considering only the one hundred micrometer particles the fallout pad results summed for the entire exposure interval of the open trays, Jan. 30 to Feb. 21, would amount to less than a thousand (MIL-STD-1246B, Level 500).

SUMMARY OF LATS PARTICULATE CLEANLINESS BASED ON TAPELIFT SAMPLES



LATS AVERAGE PARTICLE FALLOUT PER 24 HR.

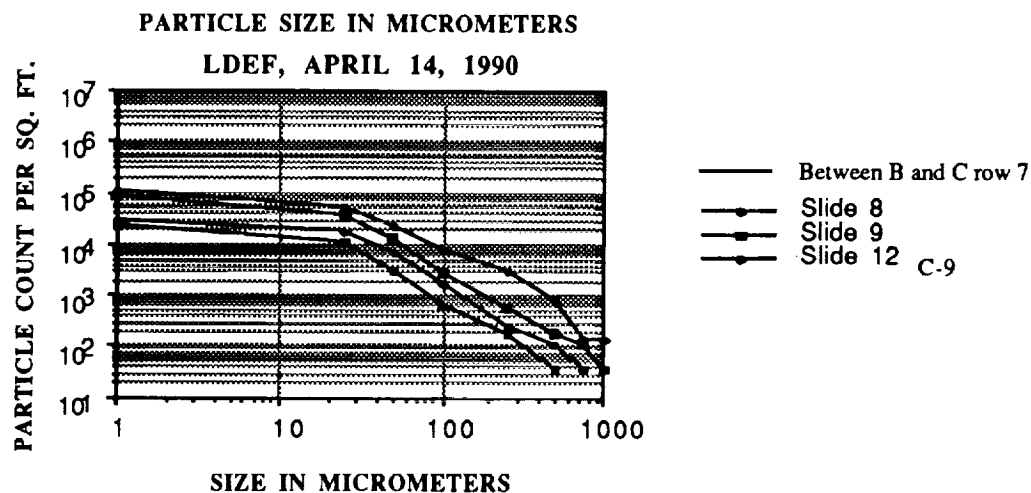
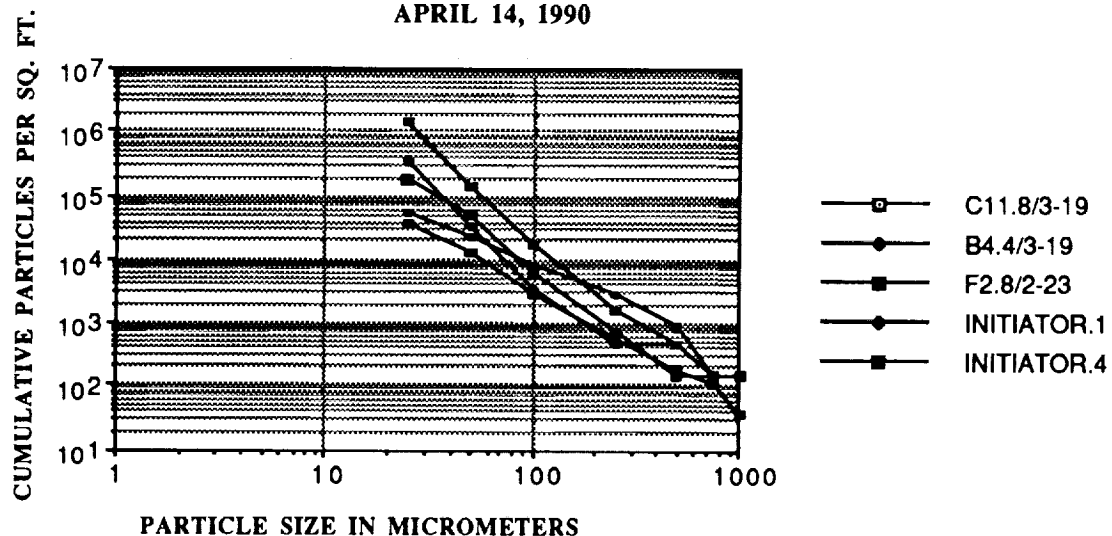
Based on Fallout Plate Samples



TAPELIFT RESULTS FOR LDEF SURFACES UNDER TRAY CLAMPS AND FROM THE INTERIOR OF LDEF

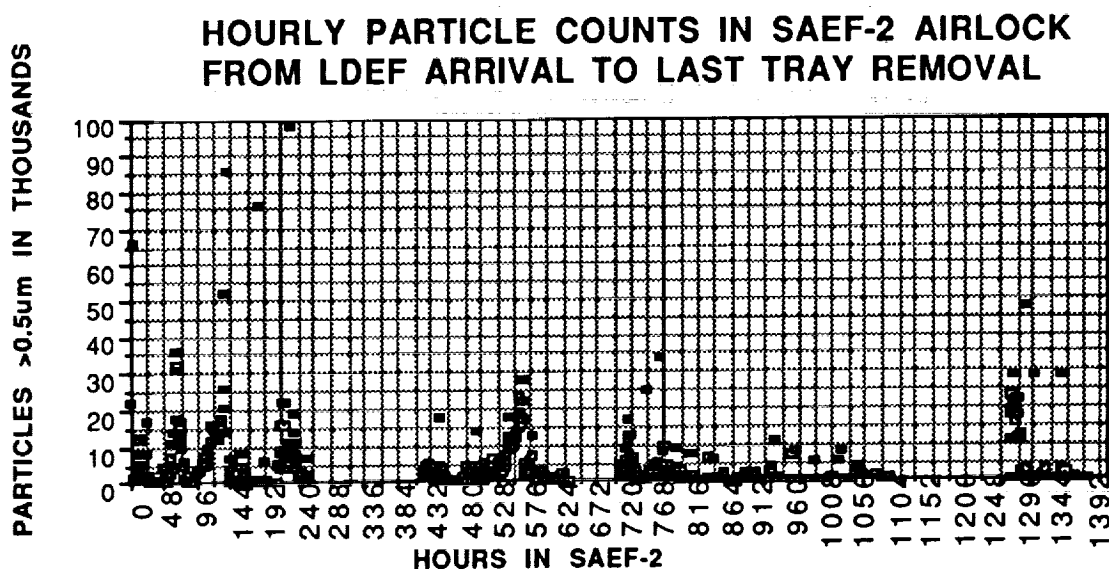
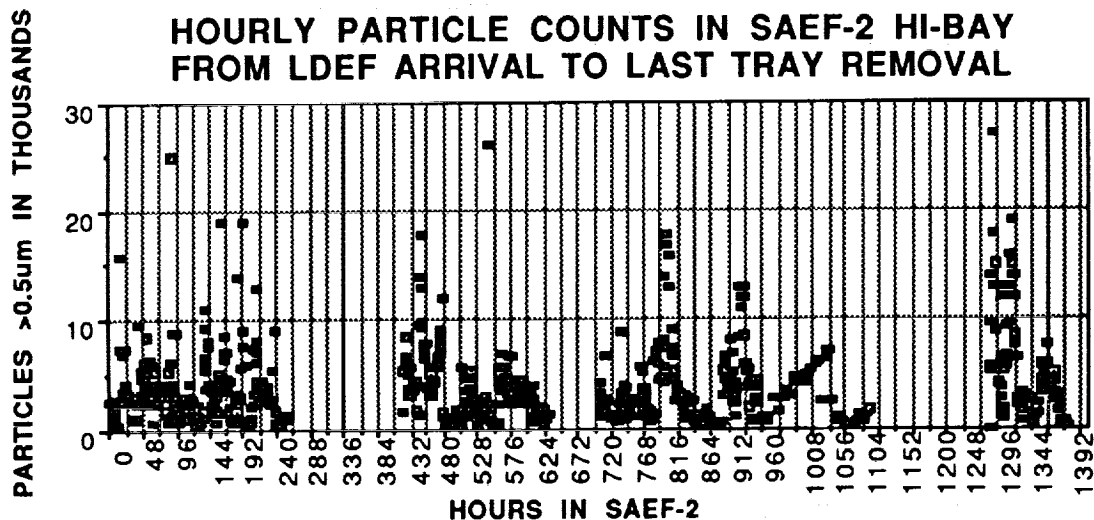
Tapelifts were collected from the surface of LDEF that had been covered with tray clamps prior to the removal of the trays. The legend indicates the tray clamp under which the sample was collected and the date on which that clamp had been removed (for example, the first entry below indicates the sample was collected from under the eighth tray clamp of tray C-11. This clamp had been removed with the tray on the 19th of March and the surface had been exposed to the SAEF-2 environment until the sample was collected on the 14th of April). It had been anticipated that the particle distribution would reflect the duration of SAEF-2 exposure. This was not the case. Although high particle counts were seen the type of particle was biased toward manufacturing and assembly residues and not so much toward the typical SAEF-2 debris. The particle population under clamp 8 of tray F-02 was about the same as that under clamp 4 of tray B-04 even though the F-02 area had been exposed in SAEF-2 for nearly three weeks longer. All of the samples from under the tray clamps were more contaminated than the tray surfaces or the other exposed surfaces of LDEF. This suggests that the tray clamps retained contaminants that were removed from other surfaces following integration. Surfaces not protected by tray clamps (INITIATOR samples and those shown in the lower chart) are nearly an order of magnitude cleaner. The contaminants on these other surfaces are also different indicating populations of the type seen on the tray surfaces.

**TAPELIFT DATA FROM LDEF SURFACE COLLECTED
APRIL 14, 1990**



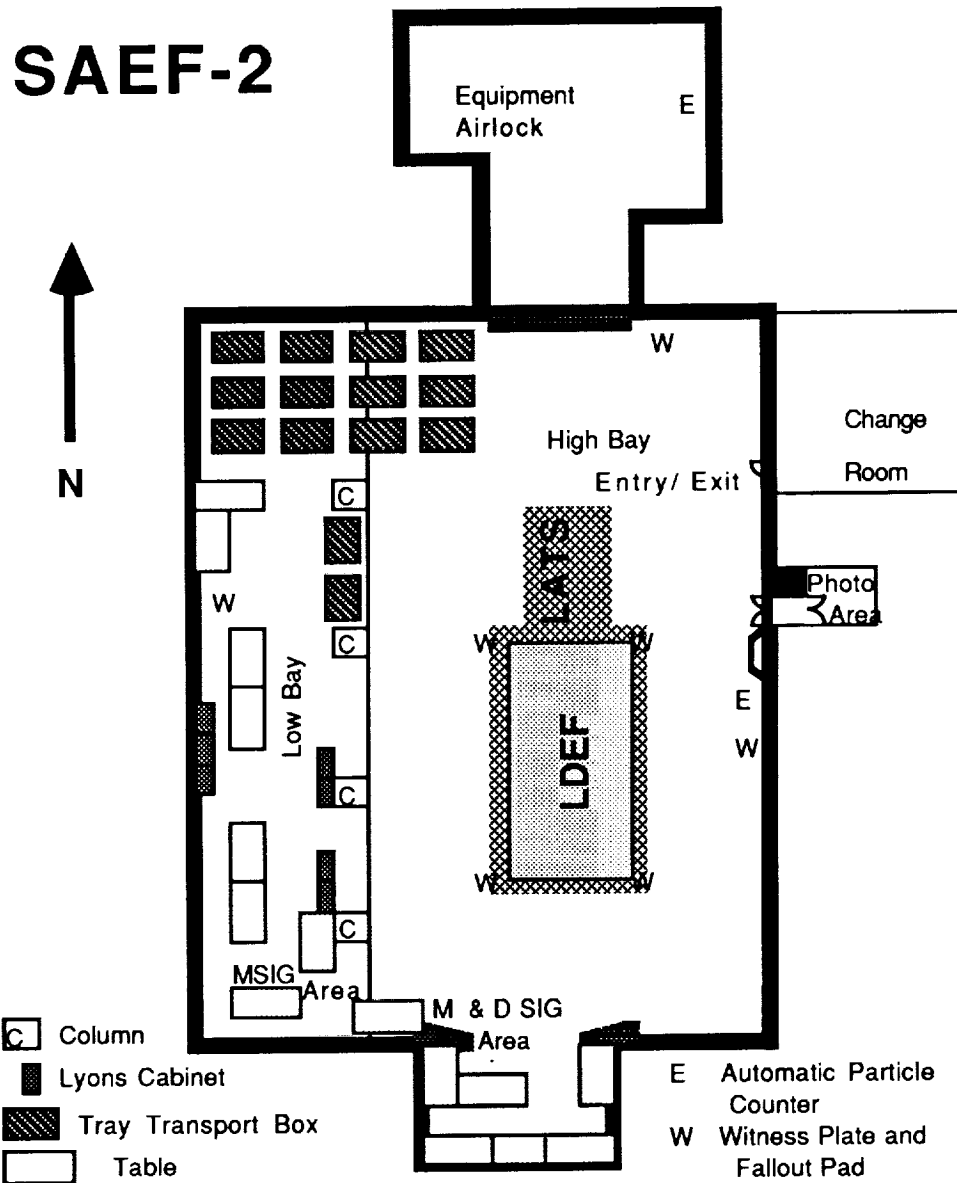
AIRBORNE PARTICLE COUNTS $>0.5 \mu\text{m}$ FOR THE SAEF-2 CLEANROOM HIGH BAY AND THE EQUIPMENT AIRLOCK

The graphs below show the particle count at each hour mark recorded during LDEF's exposure to the SAEF-2 environment. The hourly counts in the High Bay cleanroom never exceeded thirty thousand. Individual counts on one occasion exceeded one hundred thousand but that was a transient condition associated with the moving of a scaffold that was above and adjacent to the particle counter. This event lasted only a few minutes and the airborne particle count dropped back well below one hundred thousand before the next hour mark. The scaffolding was moved periodically but it was normally closer to LDEF and didn't significantly disturb the particle counter. In the Airlock the particle counts were typically higher. When materials were entering the Airlock from outside the count would exceed one hundred thousand. The particle count would recover generally within an hour. The airborne particle counts indicated that the air being supplied to SAEF-2 was being effectively scrubbed by the HEPA's. In a conventional non-laminar flow cleanroom with a single sensor mounted ten feet high on the wall relatively little information is gathered with regard to the larger particle population (five micrometers and greater).



SAEF-2 FLOOR PLAN WITH LDEF ON LATs

The locations of the wall mounted airborne particle monitors and the floor or LATs bed fallout pads are shown in this illustration.

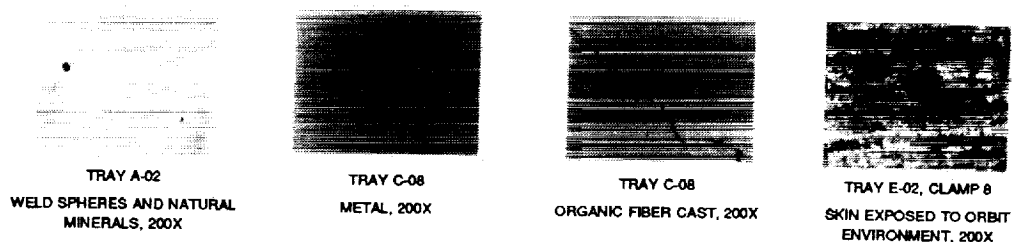


PARTICLES THAT INDICATE DEBRIS FROM TRAY ASSEMBLY AND PARTICLES THAT ARE TRACEABLE TO SPECIFIC SOURCES

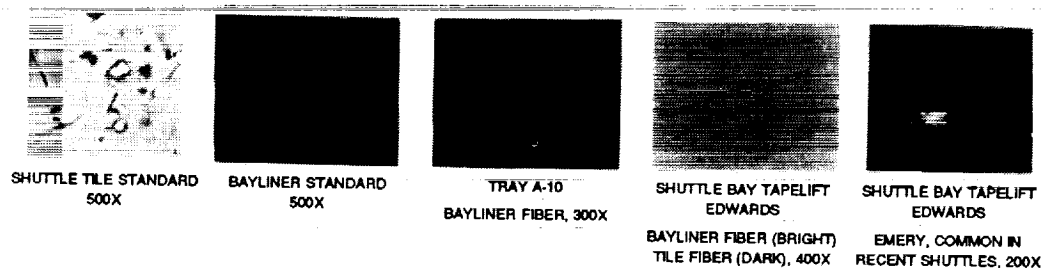
Particles that accumulated during assembly up through launch and that were present in orbit can be distinguished from more recent particles by shadow effects on the underlying surface associated with the particles. On the trailing side tray A-02 the weld sphere and the mineral particle are associated with a shadow in the deposited molecular film indicating their presence early in the mission. The wear metal particle seen on tray C-08 protected part of the surface it covered from atomic oxygen exposure during the mission. Organic particles present early in the mission also provided protection for the underlying surface but only so long as they survived the attack of atomic oxygen. When they were finally consumed the underlying surface was protected only by what ash remained. The temporary protection provided by these particles resulted in a silhouette of the particle on the surface detected as a less eroded area. Where shadow effects were not easily seen the particle itself could indicate its long term orbital exposure, such as the example of the skin cell on clamp 8 of tray E-02. These particles are all typical of residues from tray assembly operations.

The Shuttle Bay was also a source of particles. Two materials characteristic of the Shuttle are the glass fibers from the Shuttle thermal protection tiles and the Teflon coated glass particles from the liner of the Shuttle Bay. When some of the glass fibers collected in the Shuttle and on LDEF were compared to standard samples from these sources they were found to be the same.

ASSEMBLY DEBRIS TRACER PARTICLES

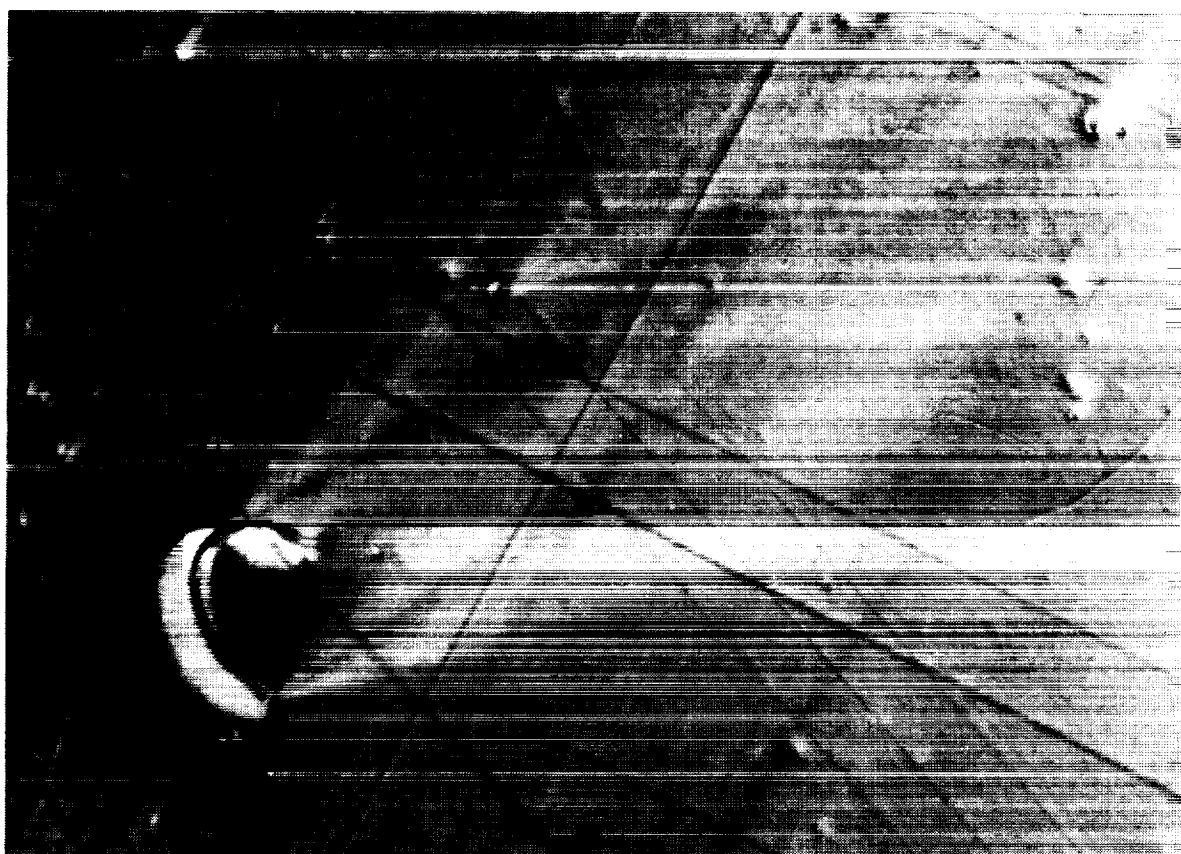


SHUTTLE BAY TRACER PARTICLES



FEATURES INDICATING A PARTICLES PRESENCE DURING ORBIT

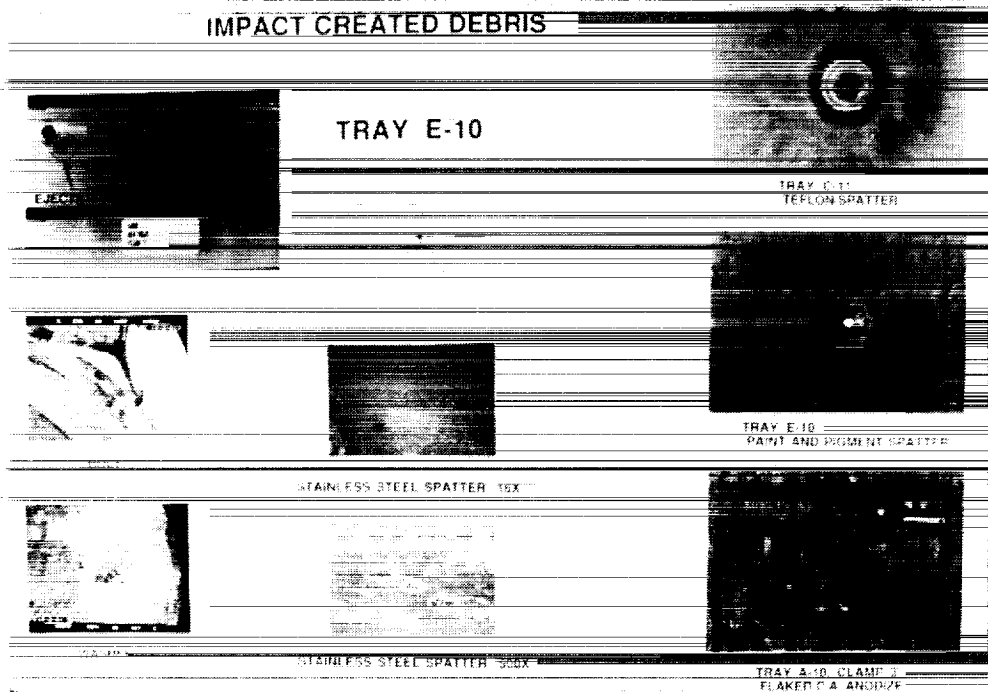
This photograph shows an area of the surface on tray A-04. Particles present during orbit have a shadow (bright area) associated with them. Particles removed after the formation of the shadow leave only the shadow to indicate their past presence (small bright spot near center of photo). The halo around each particle is believed to be the result of outgassing materials held by capillary attraction at the interface between the particle and the tray surface. The "plume" pattern is believed to be the effect of the molecular flow over the surface.



ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

PARTICLES GENERATED IN ORBIT

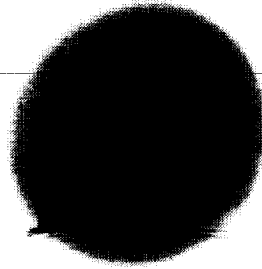
Micrometeorites or space debris impacts on the surface of LDEF created particles that could deposit on LDEF. The photographs on the left of this foil characterize one such event when a micrometeorite impacted with the side of a stainless steel bolt on tray E-10. Examples of other materials releasing particles as a result of impacts are given for Teflon on tray C-11, paint on tray E-10, and chromic acid anodize on tray A-10.



PARTICLES DEPOSITED AS LIQUID DROPLETS

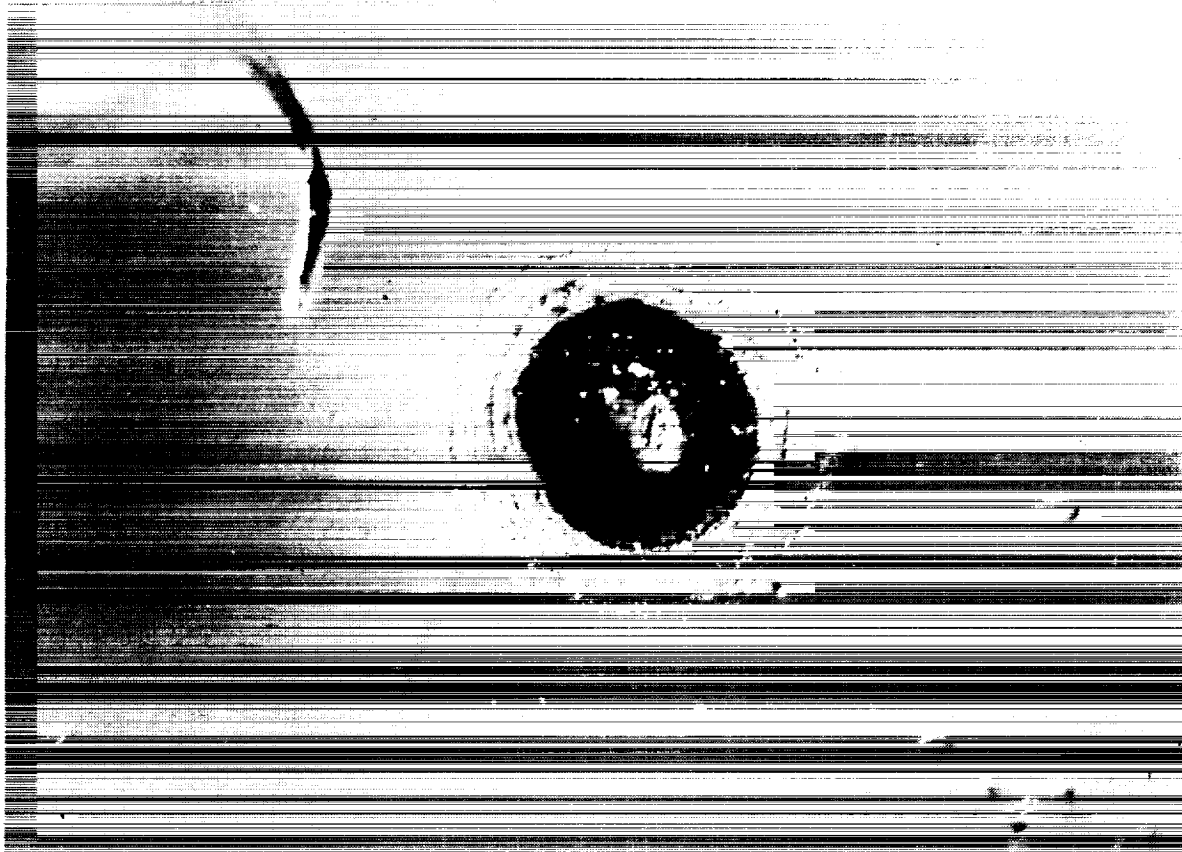
A number of brown spots were found distributed widely over the surface of LDEF. These spots were circular or globular in shape indicating the effects of surface tension on their formation. Within these deposits particles were generally distributed concentrically about the center of the droplet. This is all consistent with the deposition of liquid aerosols on the surface. There are many sources for liquid aerosols during assembly, during orbit, and following recovery.

This photograph illustrates one of at least four types of brown spots seen on LDEF. This type is characterized by a high residual material content and significant organic content. It was collected from under tray clamp number four of tray B-08 and had been deposited on the frame of LDEF prior to the integration of the experiment trays.



PARTICLES DEPOSITED AS LIQUID DROPLETS

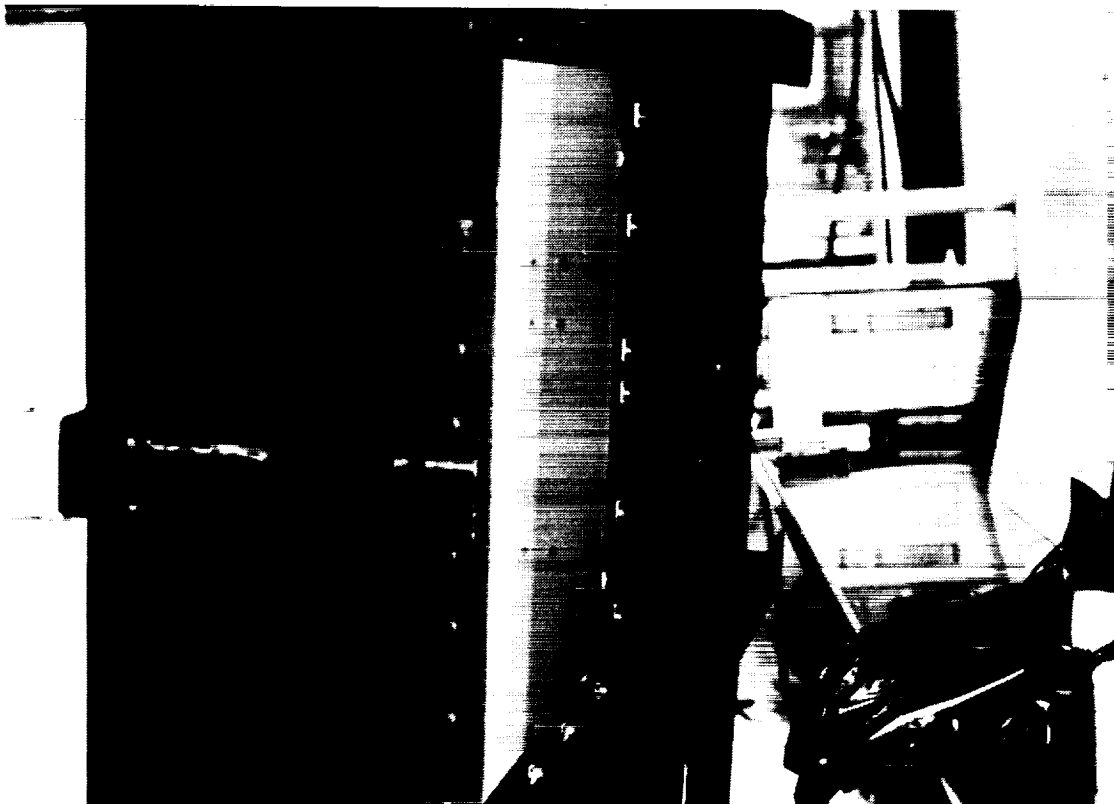
This type of brown spot is characterized by concentric rings of particles outside of the central deposit. Skin cells are common in this droplet. This is typical of "sneeze" type residues deposited before orbital exposure. This droplet was photographed in SAEF-2 and was found on the surface of experiment A0187-2, tray C-03.



ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

MOLECULAR FILM DEPOSITED ON INTERIOR SIDE OF TRAY F-06

Molecular films were visually detected either by the brown discoloration seen on light surfaces or the thin film interference colors caused by them on black surfaces. This photograph illustrates the interference color effect* seen on the ram facing side panel of tray F-06. Each red band beginning with the brown-red near the edge just before the first blue band corresponds to a thickness of approximately 100 nanometers (0.1 micrometers) added to the film's thickness. Notice the continuation of the pattern on the next brace.

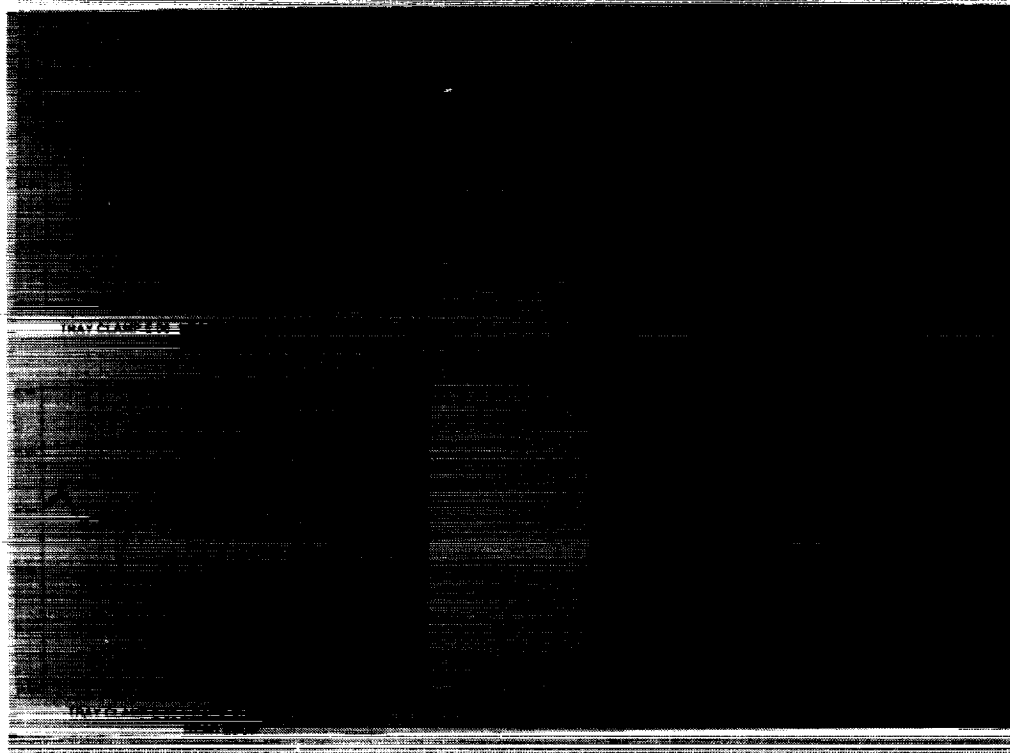


ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

*Shown in black and white only.

TYPICAL INFRARED SPECTRA OF MOLECULAR DEPOSIT

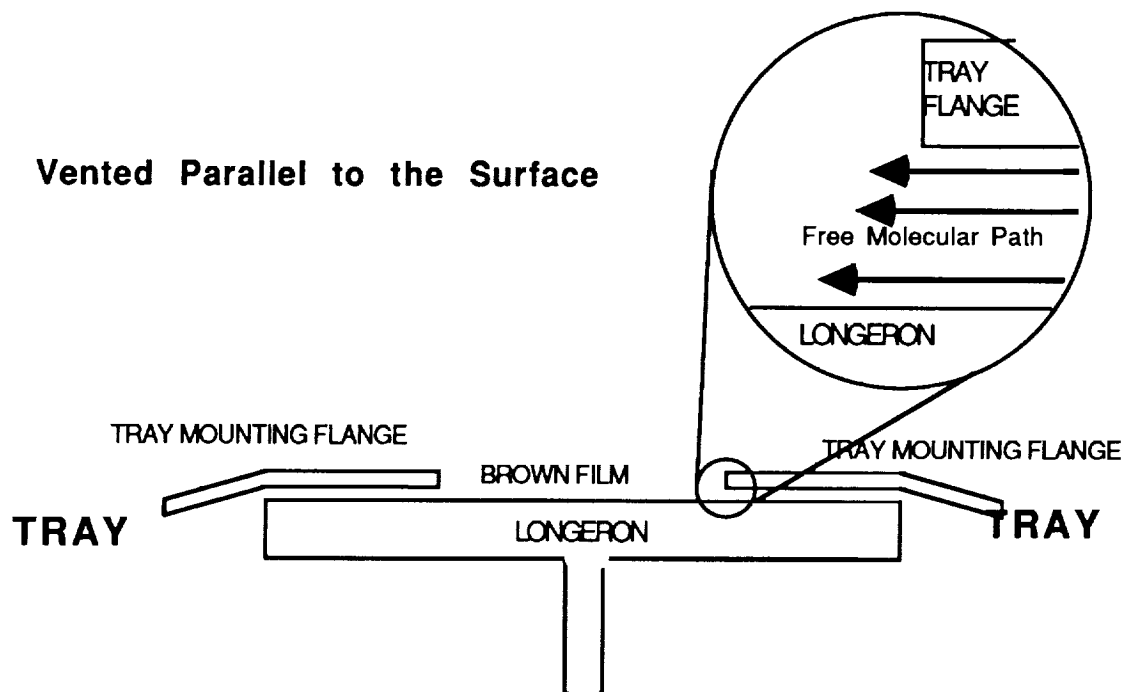
The infrared spectra of the brown film from most locations were remarkably similar. On the right of this foil are the spectra from the earth end frame of LDEF and from the space end frame. The same basic functional groups are indicated in similar proportions. The spectra on the left side are an example of the organic materials detected as residues between the tray clamps and shims on LDEF.



VENT PATH FROM INTERIOR OF LDEF ALONG THE EDGE OF TRAYS

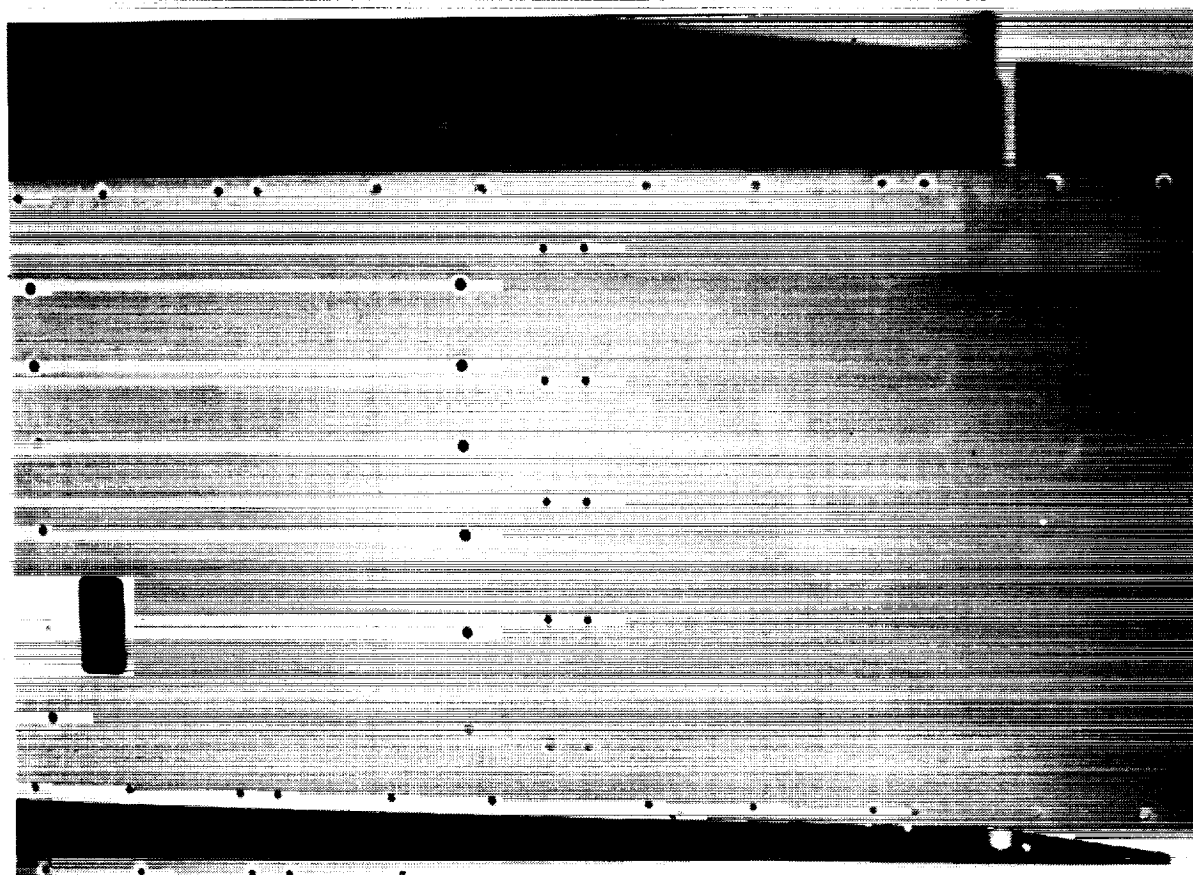
Many of the vent paths on LDEF consisted of narrow openings between parallel plates of metal. The edge of the trays are an example of such a path. Molecules escaping from the interior along such a path would tend to parallel the surface of LDEF. Any encounter with another molecule would have a fifty percent probability of directing the molecule toward the surface of LDEF. This may help explain the relatively high deposition efficiency exhibited by the exterior surface of LDEF.

VCM Vented Parallel to the Surface



HANDPRINT ON TRAY F-06

On the tan stained surface of the floor of tray F-06 a lighter colored pattern could be seen. This pattern is a palm print. The trays were handled without gloves and while working with this tray it began to tip. One of the individuals handling the tray put up his hand to stop the tray. The cleanliness requirements for LDEF didn't require a control of surface organics or particles that were not obvious to the unaided eye so no attempt at wiping the tray clean was made. This pattern is of interest for two reasons. First, it illustrates the conditions under which LDEF was assembled. Second, it creates questions regarding the mechanism that turned the tray floor tan where it was "cleaner" but not where it had been contacted by a bare hand. In some other areas fingerprints were seen that had turned black from exposure to ultraviolet light.



ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH